इंटरनेट मानक

Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

"जानने का अधिकार, जीने का अधिकार"
Mazdoor Kisan Shakti Sangathan
"The Right to Information, The Right to Live"

"पुराने को छोड नये के तरफ"
Jawaharlal Nehru
"Step Out From the Old to the New"

IS 2632 (1964): Crotonaldehyde [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

"ज्ञान से एक नये भारत का निर्माण"
Satyanarayan Gangaram Pitroda
"Invent a New India Using Knowledge"

Act Number 22 of 2005
The Indian Parliament
RIGHT TO INFORMATION

Made Available By
Public.Resource.Org

"ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है"
Bhartṛhari—Nītiśatakam
"Knowledge is such a treasure which cannot be stolen"

BLANK PAGE

BRUTUM FULMEN
IGNORANTIA JURIS NON EXCUSAT
PROTECTED BY COPYRIGHT

IS : 2632 - 1964

Indian Standard

SPECIFICATION FOR CROTONALDEHYDE

First Reprint AUGUST 1976
(Incorporating Amendment No. 1)

UDC 661·727·3

© *Copyright* 1976

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

June 1964

Indian Standard

SPECIFICATION FOR CROTONALDEHYDE

Alcohols and Allied Products Sectional Committee, CDC 2

Chairman	*Representing*
Shri G. Gundu Rao	All India Distillers' Association, New Delhi
Members	
Shri R. C. Aitken	E. I. D. Parry Ltd., Nellikuppam
Shri T. K. Rangaswami (*Alternate*)	
Dr V. R. S. Arni	Union Carbide India Ltd., Calcutta
Dr H. C. Bijawat (*Alternate*)	
Shri B. B. Bagalkot	Indian Chemical Manufacturers' Association, Bombay
Dr A. H. Amin (*Alternate*)	
Shri V. J. Bakre	Central Revenues Control Laboratory (Ministry of Finance)
Shri D. K. Sankaran (*Alternate*)	
Shri A. M. Curle	Carew & Co Ltd., Calcutta
Shri W. R. Damle	Government of Uttar Pradesh
Shri M. A. Ekambaram	The Sirsilk Limited, Sirpur-Kaghaznagar
Shri T. S. Modi (*Alternate*)	
Dr B. G. Gunde	The Tata Oil Mills Co Ltd., Bombay
Dr (Miss) B. S. Khambata (*Alternate*)	
Shri O. P. Gupta	Directorate General, Ordnance Factories (Ministry of Defence), Calcutta
Dr V. M. I. Nambissan (*Alternate*)	
Shri G. Narasimha Iyengar	The Mysore Sugar Co Ltd., Bangalore
Shri K. Lakkappa (*Alternate*)	
Shri S. Krishnamurty	Indian Chemical Manufacturers' Association, Calcutta
Dr S. Rohatgi (*Alternate*)	
Shri B. Narayan	The Hindusthan Sugar Mills Ltd., Golagokarannath
Shri N. B. Sen Gupta	Directorate General of Supplies & Disposals
Shri K. L. Banerjee (*Alternate*)	
Dr K. K. G. Sikchi	The Alkali & Chemical Corporation of India, Ltd., Calcutta
Dr S. Ganguly (*Alternate*)	
Shri L. R. Sud	Ministry of Defence (R & D)
Shri A. P. Chakraverty (*Alternate*)	
Dr S. N. Tewari	Directorate of Medical and Health Services, Government of Uttar Pradesh
Shri D. M. Trivedi	Synthetics & Chemicals Ltd., Bombay
Shri P. R. Mahalingam (*Alternate*)	

(*Continued on page* 2)

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

(*Continued from page* 1)

Members	*Representing*
SHRI S. L. VENKITESWARAN	Directorate General of Technical Development
DR SADGOPAL, Deputy Director (Chem)	Director, ISI (*Ex-officio Member*)

Secretaries

SHRI D. DAS GUPTA
Assistant Director (Chem), ISI

SHRI M S SAXENA
Extra Assistant Director (Chem), ISI

Crotonaldehyde Panel, CDC 2 : P7

Convener	
SHRI M. A. EKAMBARAM	The Sirsilk Ltd., Sirpur-Kaghaznagar
Member	
DR H. C. BIJAWAT	Union Carbide India Ltd., Calcutta

Indian Standard

SPECIFICATION FOR CROTONALDEHYDE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 15 January 1964, after the draft finalized by the Alcohols and Allied Products Sectional Committee had been approved by the Chemical Division Council.

0.2 Crotonaldehyde is a useful chemical intermediate by virtue of its highly reactive character. It undergoes the characteristic reactions of the ethylenic double bond as well as those of simple aldehydes. It is a starting material for a wide range of aldehyde-amine type of rubber antioxidants and accelerators; for example, crotonic acid, butyraldehyde and butanol, quinoline yellow and other dyes and for certain synthetic resins. It is also used as a denaturant for industrial alcohol. The production of crotonaldehyde has been recently established in the country. This standard is expected to assist manufacturers in regulating the quality of material produced and the consumers in selecting a material of acceptable quality for use as a denaturant for industrial alcohol.

0.3 Wherever a reference to any Indian Standard appears in this specification, it shall be taken as a reference to the latest version of the standard.

0.4 This standard is one of a series of Indian Standards on alcohols and allied products.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS:2-1960 Rules for Rounding Off Numerical Values (*Revised*). The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

0.6 This standard is intended chiefly to cover the technical provisions relating to the supply of crotonaldehyde, and it does not include all the necessary provisions of a contract.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of test for crotonaldehyde suitable for use as a denaturant for industrial alcohol.

2. REQUIREMENTS

2.1 Description – The material shall be a clear liquid with a pungent suffocating odour consisting essentially of crotonaldehyde. The material shall be free from sediment and suspended matter.

2.2 The material shall also comply with the requirements given in Table I.

TABLE I REQUIREMENTS FOR CROTONALDEHYDE

Sl No.	Characteristic	Requirement	Method of Test (Ref to Cl No. in Appendix A)
(1)	(2)	(3)	(4)
i)	Specific gravity at 27°/27°C, *Max*	0·872	A-1
ii)	Aldehyde content (as crotonaldehyde), percent by weight, *Min*	85	A-2
iii)	Acidity (as CH_3COOH), percent by weight, *Max*	0·3	A-3

3. PACKING AND MARKING

3.1 Packing

3.1.1 The material shall be packed in suitable containers as agreed to between the purchaser and the supplier, and subject to the provisions of Red Tariff No. 18 Rules and Rates for Conveyance by Rail of Explosives and Other Dangerous Goods, issued by the Indian Railway Conference Association with any alterations or additions made thereafter.

3.1.2 All containers in which the material is packed shall be dry and clean so that no impurities harmful to the end use of the material are introduced.

3.2 Marking

3.2.1 The material shall be supplied in accordance with the marking and delivery instructions given by the purchaser.

3.2.2 *Each container shall be marked with:*

a) name of the material;
b) manufacturer's name, initials or trade-mark, if any;
c) net, gross and tare weight; and
d) month and year of manufacture.

3.2.3 Each container may also be marked suitably with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Preparation of Test Samples — Representative samples of the material shall be drawn as prescribed in Appendix B.

4.2 Number of Tests — Tests for the determination of all the characteristics specified shall be conducted on the composite sample.

4.3 Criteria for Conformity — The material shall be taken as conforming to this specification, if the test results of the composite test sample satisfy all the requirements prescribed.

5. TEST METHODS

5.1 Tests shall be conducted according to the methods prescribed in Appendix A. References to relevant clauses of Appendix A are given in col 4 of Table I.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water [*see* IS : 1070-1960 Specification for Water, Distilled Quality (*Revised*)] shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis

APPENDIX A

[*Table* I, *Items* (i) *to* (iii)]

METHODS OF TEST FOR CROTONALDEHYDE

A-1. DETERMINATION OF SPECIFIC GRAVITY

A-1.1 Apparatus

A-1.1.1 *Specific Gravity Bottle* — 50-ml capacity of Regnault type.

A-1.2 Procedure — Clean, dry and weigh the specific gravity bottle and the stopper. Fill the bottle with water and immerse it up to the neck in a constant temperature bath maintained at 27·0 ±0·2°C for 20 minutes and keep at this temperature for additional 20 minutes. Suck off the water with a bit of filter paper till the level reaches the graduation mark and weigh again. Empty the bottle, clean and dry. Repeat the operation with the material at 27°C.

A-1.3 Calculation

$$\text{Specific gravity at } 27^\circ/27^\circ\text{C} = \frac{A - B}{C - B}$$

where

A = weight, in g, of the specific gravity bottle with the material at 27°C;

B = weight, in g, of the specific gravity bottle; and

C = weight, in g, of the specific gravity bottle with water at 27°C.

A-2. DETERMINATION OF ALDEHYDE CONTENT

A-2.1 Reagents

A-2.1.1 *Alcoholic Solution of Hydroxylamine Hydrochloride*, 0·5 *N* — Dissolve 35 g of hydroxylamine hydrochloride in 150 ml of distilled water and dilute to one litre with methanol.

A-2.1.2 *Aqueous Solution of Triethanolamine*, 0·5 *N* — Transfer 65 ml (74 g) of 98 percent triethanolamine to a 1 000 ml volumetric flask and dilute to the mark with water.

A-2.1.3 *Standard Sulphuric Acid Solution (Dilute)* — 0·5 N.

A-2.1.4 *Bromophenol Blue Indicator* — 0·04 percent (*w/v*) alcoholic solution.

A-2.2 Procedure

A-2.2.1 *Preparation of Neutral Alcoholic Solution of Hydroxylamine Hydrochloride* — Add 30 ml of alcoholic solution of bromophenol blue indicator to one litre of the hydroxylamine hydrochloride reagent. From a burette add triethanolamine solution until the solution appears greenish-blue by transmitted light. Prepare the solution fresh before each series of the analysis.

A-2.2.2 Prepare a sufficient number of heat-resistant pressure bottles to make all the blank and sample determinations in duplicate. To each bottle add 65 ml of neutralized alcoholic solution of hydroxylamine hydrochloride and from a burette add exactly 50 ml of triethanolamine solution. Before capping, purge the bottle for 2 minutes with a gentle stream of nitrogen. This is best accomplished by means of a glass tube inserted through the neck of the bottle and clamped so that the opening is just above the surface of the liquid. Reserve two of the bottles for blanks and into each of the other bottles introduce about one gram of the sample weighed to the nearest 0·1 mg. Allow the samples to stand together with the blanks at room temperature for 60 minutes. Shake the bottles occasionally. Cool each of the bottle slightly with tap water and uncap carefully to prevent loss of contents. Allow the contents to return to room temperature and titrate each of the blanks with standardized dilute sulphuric acid to greenish-blue end-point. Titrate each of the samples with standardized dilute sulphuric acid to the colour of the blanks approaching the end point dropwise until the colours match by transmitted light.

A-2.3 Calculation

Aldehyde content (as crotonaldehyde), percent by weight $= \frac{(B - A) \times 7{\cdot}00 \times N}{W}$

where

B = average volume in ml of standard sulphuric acid solution required for the blanks,

A = volume in millilitres of standard sulphuric acid solution required for the sample,

N = normality of standard sulphuric acid solution, and

W = weight in g of sample taken for the test.

A-3. DETERMINATION OF ACIDITY

A-3.1 Reagents

A-3.1.1 *Rectified Spirit* — 95 percent [conforming to IS : 323-1959 Specification for Rectified Spirit (*Revised*)].

A-3.1.2 *Standard Sodium Hydroxide Solution* — 0·1 N.

A-3.1.3 *Phenolphthalein Indicator* — Dissolve 0·5 g of phenolphthalein in 60 ml of rectified spirit [conforming to IS : 323-1959 Specification for Rectified Spirit (*Revised*)] diluted with water to 100 ml. Make the solution faintly pink by adding dilute sodium hydroxide solution.

A-3.2 Procedure — Depending on the degree of acidity, choose a suitable quantity of the material. Dissolve 10 ml of the material in 50 ml of rectified spirit, neutralized to phenolphthalein and titrate the mixture with standard sodium hydroxide solution using 0·5 ml of the phenolphthalein indicator.

A-3.3 Calculation

$$\text{Acidity (as acetic acid), percent by weight} = \frac{6\,V\,N}{W}$$

where

V = volume, in ml, of standard sodium hydroxide solution;
N = normality of standard sodium hydroxide solution; and
W = weight, in g, of the material taken for the test.

APPENDIX B

(*Clause* 4.1)

SAMPLING OF CROTONALDEHYDE

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall be taken in a protected area with good ventilation.

B-1.2 Sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by shaking, stirring, rolling or by any other suitable means.

B-1.5 The samples shall be placed in suitable, clean, dry and air-tight, amber-coloured glass or metal containers on which the material has no action.

B-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.7 Each sample container shall be sealed air-tight with a suitable stopper after filling and marked with the manufacturer's name or trade-mark, the month and year of manufacture of the material, the batch number (if available), and other details of sampling, such as the date of sampling, sampler's name, etc.

B-2. SAMPLING INSTRUMENT

B-2.1 It is made of thick glass or metal on which the material has no action and is 20 to 40 mm in diameter and 400 to 800 mm in length (*see* Fig. 1).

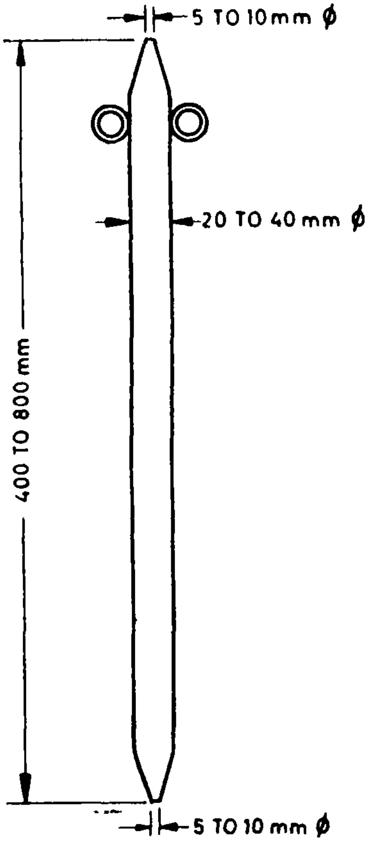

FIG. 1 SAMPLING TUBE

The upper and lower ends are conical and reach 5 to 10 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper end. For drawing samples, the apparatus is first closed at the top with the thumb or a stopper and lowered till a desired depth is reached. It is then opened for a short time to admit the material at the desired depth and finally closed and withdrawn.

B-2.1.1 For small containers, the size of the sampling tube may be altered suitably.

B-3. SCALE OF SAMPLING

B-3.1 Lot — In any consignment, all the containers of the same size and drawn from the same batch of manufacture shall constitute a lot. If a consignment is known to consist of different batches of manufacture or of different sizes of containers, the containers belonging to the same batch and size shall be grouped together and each such group shall constitute a separate lot.

B-3.2 For ascertaining the conformity of the material in a lot to the requirements of the specification, tests shall be carried out for each lot separately. The number of containers to be selected for this purpose from a lot shall depend on the size of the lot and shall be in accordance with the col 1 and 2 of Table II.

TABLE II SCALE OF SAMPLING

LOT SIZE N (1)	NO. OF CONTAINERS TO BE SELECTED n (2)
Up to 3	*
4 „ 15	3
16 „ 40	4
41 „ 65	5
66 „ 110	7
111 „ 180	8
181 „ 300	9
301 and above	10

*When the lot size is 3 or less, the number of containers to be selected and the method of judging the conformity of the lot to the specification requirements shall be as agreed to between the purchaser and the supplier.

B-3.3 These containers shall be selected at random from the lot and in order to ensure randomness of selection a random number table as agreed to between the purchaser and the supplier shall be used. In case such a table is not available, the following procedure is recommended for use:

> Arrange all the containers in the lot in a systematic manner and starting from any container, count them as 1, 2, ... up to r and so on, where r is the integral part of N/n (N being the lot size and n the number of containers to be selected). Every rth container thus counted shall be withdrawn to give a sample for tests.

B-4. PREPARATION OF TEST SAMPLES

B-4.1 From each of the containers selected according to **B-3.3**, a small representative portion of the material sufficient for carrying out tests as indicated in **2** shall be drawn with the help of the sampling tube (*see* **B-2.1**).

B-4.2 Out of these portions, equal quantity of material shall be taken and mixed thoroughly to form a composite sample, not less than 1 500 ml The composite test sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample

B-4.3 The composite samples shall be transferred to containers of 600-ml capacity and shall be sealed and marked with full identification particulars given in **B-1.7**

B-4.4 The referee test sample shall also bear the seal of both the purchaser and the supplier It shall be kept at a place agreed to between the purchaser and the supplier, to be used in the case of any dispute between the two

PUBLICATIONS OF INDIAN STANDARDS INSTITUTION

INDIAN STANDARDS

Over 8 500 Indian Standards covering various subjects have been issued so far. Of these, the standards belonging to the Chemical Group fall under the following categories:

Acids
Adhesives
Alcohols and allied products
Alkalis
Brushware
Ceramicware, enamelware and laboratory porcelain
Chemical hazards and safety
Chemicals, inorganic (miscellaneous)
Chemicals, organic (miscellaneous)
Coal and coke
Coal carbonization products
Coated fabrics
Cosmetics and toilet goods
Dental material
Drying oils
Dye intermediates
Electroplating chemicals
Explosive and pyrotechnic materials
Fertilizers
Fillers, stoppers and putties
Footwear
Glass and glassware
Industrial gases
Inks and allied products
Laboratory glassware thermometers and related apparatus
Lac and lac products
Leather, leather goods and leather dressings
Linters and allied products
Lubricants and related products
Oil pastes
Oils & fats, oleaginous seeds and fruits
Painters' materials (miscellaneous)
Paper and its products
Paper and pulp board packaging materials
Perfumery materials, natural and synthetic
Petroleum and petroleum products
Photographic chemicals
Pigments and extenders
Plastics
Polishes
Printing inks
Ready mixed paints and enamels
Rubber and rubber products
Soaps and other surface active agents
Tanning materials and allied products
Thermal insulation materials
Thinners and solvents
Varnishes and lacquers
Water and water treatment
Water based paints
Unclassified

OTHER PUBLICATIONS

ISI Bulletin (Published Every Month)	
Single Copy	Rs 4·00
Annual Subscription	Rs 36·00
Standards : Monthly Additions	
Single Copy	Re 0·30
Annual Subscription	Rs 3·00
Annual Reports (from 1948-49 Onwards)	Rs 2·00 to 6·00
ISI Handbook, 1975	Rs 30·00

INDIAN STANDARDS INSTITUTION

Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002

Telephone : 27 01 31 (20 lines) **Telegrams : Manaksanstha**

Regional Offices:

			Telephone
Western	Novelty Chambers, Grant Road	BOMBAY 400007	37 97 29
Eastern	5 Chowringhee Approach	CALCUTTA 700072	23-08 02
Southern	C.I.T. Campus, Adyar	MADRAS 600020	41 24 42

Branch Offices:

'Pushpak', Nurmohamed Shaikh Marg, Khanpur	AHMADABAD 380001	2 03 91
'F' Block, Unity Bldg, Narasimharaja Square	BANGALORE 560002	2 76 49
Ahimsa Bldg, SCO 82-83, Sector 17C	CHANDIGARH 160017	2 83 20
5-8-56/57 Nampally Station Road	HYDERABAD 500001	4 57 11
117/418 B Sarvodaya Nagar	KANPUR 208005	82 72
B C.I. Bldg (3rd Floor), Gandhi Maidan East	PATNA 800004	5 36 55
Hantex Bldg(2nd Floor), Rly Station Road	TRIVANDRUM 695001	32 27

Printed at Today & Tomorrow's Printers & Publishers, New Delhi, India